2.4 Safety Procedures

General laboratory procedures were followed, as outlined in the Standard Operating Procedures (SOP) for Research and the University of Akron Safety Manual under the guidance of Christopher M. Miller Ph.D., PE, and Allen L. Sehn Ph.D., P.E., the principle investigators at the University of Akron. The University of Akron has undergone a compliance inspection by the INEEL. All waste materials, including excess grout-organic sludge, were temporarily stored (i.e. less than 1 week) in Room 6 ASEC until Dr. Song was notified. Once Dr. Song was contacted, all waste materials were moved to Room 133 in the Knight Chemical Laboratory (KNCL). Room 133 is a 90-day storage area. Waste in Room 133 is disposed of monthly by two companies: (1) Chemical Analytics Inc. (Romulus, MI) and (2) Envirocure (Pittsburgh, PA). Chemical Analytics is a broker from Romulus, MI using Petrochem to fuel blend the majority of the waste and incinerate it in Minnesota. Envirocure utilizes WTI for incineration and EEI for chemical neutralization.

3. RESULTS AND DISCUSSION

The results and discussion for this project are presented in six individual sections as follows: 1) Implementability Testing, 2) Interference Tolerance Testing, 3) Physical Testing, 4) Chemical Testing, 5) WAXFIX Boron Distribution, and 6) Grout-Organic Sludge Encapsulation.

3.1 Implementability Testing

The primary objectives of the implementability testing were to ensure that each of the grout systems meets the implementability criteria established by INEEL. For the proprietary grout systems, the materials were tested as received from the suppliers. For the non-proprietary grout systems, the formulations were modified in an effort to produce grout formulations that satisfy the implementability criteria and are likely to perform well in the remainder of the testing program.

The results of the implementability tests are presented in Table 5. There was no acceptance criterion established for the specific gravity of the grout. The specific gravity values of the five grouts ranged from 1.60 for S grout to 2.16 for T grout.

The results of the Marsh funnel test provide an indication of the viscosity of the grouts shortly after mixing. A lower marsh funnel time indicates lower viscosity, and a higher marsh funnel time indicates higher viscosity. The screening criterion was that the Marsh funnel time must be less than 420 seconds. All of the grout mixes satisfied this criterion by a wide margin. The marsh funnel times ranged from 56 seconds for the C75 grout to 165 seconds for the E grout.

The initial thickening of the grouts was evaluated using a laboratory vane shear device to monitor the shear strength of the grouts as they changed from the fluid state to a solid state. The time required to reach a shear strength of 100 Pascals is referred to as the initial gelation time, and the time required to reach a shear strength of 1000 Pascals is referred to as the final gelation time. The acceptance criteria for both of the gelation times were set at 2 hours. With the exception of the Salt Stone grout, all of the grouts satisfied both of the gelation time requirements. The initial gelation time for the Salt Stone grout was 1.8 hours, which is 10 percent earlier than the 2-hour criterion. This grout exhibits thixotropic characteristics at early age, and if agitated, the strength would develop more slowly. Under field conditions, the grout is usually agitated during the period prior to injection by the jet grouting system. As a result, the fact that the initial gelation time is slightly below the 2-hour requirement was not considered a critical issue, and the grout was continued in the testing program. The stability of the grout refers to how well the solids stay in suspension in the grout and how well the grout retains its water. The pressure filtration test can be used to get an indication of the stability of the grout. The results of the pressure filtration test are expressed as the pressure filtration coefficient with units of the inverse of the square root of time. Lower values indicate grouts that are more stable. The specified screening criterion was stability numbers in the range from 0.1 to 0.6. The stability numbers for all of the grouts tested are below the lower end of the specified range. This indicates that all of the grouts are more stable than what was required by the acceptance criterion.

For each grout, the temperature rise during the hydration process was monitored using a thermocouple placed in the center of a sample placed in an insulated container. To meet the acceptance criterion, the peak temperature must be less than 100 degrees Celsius. All of the grouts satisfied the maximum set temperature criterion. The maximum set temperatures ranged from 28° C for the S grout to 62° C for the T grout.

There was no specific limit set for the settlement/shrinkage measurement. This measurement is another indication of the ability of the grout to retain its water during the period prior to hardening. The values were obtained by measuring the height change of specimens cast into 3-inch-diameter by 6-inch-tall cylindrical molds. The settlement is expressed as a percentage of the original height of the specimen. The shrinkage values ranged from 0.25 percent for the S grout to 3.16 percent for the E grout.

All of the grouts satisfied each of the implementability criteria with the exception of the Salt Stone grout having an initial gelation time that was slightly less than the 2-hour criterion. As discussed earlier, this is not a critical issue since the material has thixotropic properties, and under field conditions with slight agitation during the holding period, the grout would satisfy the initial gelation criterion. Based on the results of the implementability testing, all of the grouts were continued into the remaining stages of the study.

Table 5. Implementability test results and screening criteria.

	Grout Product					
	C75	E	S	T	U	
Grout Property	Modified Tank Closure	Enviro-Blend	Salt Stone	Tect HG	US Grout	Screening Criteria
Specific Gravity	1.84	1.78	1.60	2.16	1.65	
Viscosity (Marsh Funnel Time) (sec.)	56	165	110	113	58	< 420
Initial Gelation Time (hours)	4.9	9.4	1.8	6.0	4.7	> 2
Final Gelation Time (hours)	10.7	27.5	8.3	17.9	7.6	> 2
Pressure Filtration Coefficient ($min^{-0.5}$)	0.072	0.077	0.023	0.008	0.033	0.1 to 0.6
Maximum Set Temperature (deg. C)	59	32	28	62	46	< 100
Settlement/Shrinkage (%)	1.82	3.16	0.25	0.44	0.84	

In addition to these implementability tests, tests were also performed to evaluate incorporating clay dispersant into the grouts and to evaluate the change in the Marsh funnel test result over time. To evaluate the influence of incorporating clay dispersant into the grouts on the compressive strength of the grouts, specimens of each grout were prepared as usual, and clay dispersant (Master Builders PS1158) was added at the rate of 0.625% by weight of the grout. This dosage was established based on the typical dosage suggested by the manufacturer's representative, the clay content of the INEEL soil, and a soil to grout mixing ratio of 50:50 by weight. The specimens were neat grout plus clay dispersant. No soil was incorporated into the mixtures. The specimens were cured for 14 days in the concrete moist-curing room. On the 14th day, the specimens were demolded for compressive strength testing. Of the five grouts, only the US Grout specimens had gained sufficient strength to be demolded without damaging them. The US Grout specimens exhibited extensive cracking in the outer portion of the specimen. The cracks penetrated to a depth of about 6 to 8 millimeters and had an average spacing of about 1 centimeter.

Based on conversations with the manufacturer’s representatives, the clay dispersant is known to act as a strong retarding agent in cement-based grouts. In addition, like many retarding agents used in concrete, when the effects of the retardation wears off, the hydration of the cement occurs at a rate that is significantly faster than normal. As a result, the heat associated with the hydration of the cement is generated more rapidly, and the specimen will generally reach a higher temperature. In the case of the US Grout specimens, this elevated temperature may have resulted in drying of the specimen even though it was being cured in a room maintained at 100% relative humidity and about 23 degrees Celsius.

The manufacturer's representative for the clay dispersant suggested that the retardation effects would be reduced if soil were included in the mixture. The logic is that if clay particles are present, the clay dispersant will primarily act on the clay particles, and the retardation effect on the cement component will be diminished. Additional testing to confirm this theory is beyond the scope and budget of the current project.

To evaluate the change in the Marsh funnel test results for the grouts as a function of time after mixing, the grouts were prepared using the usual mixing technique, and the grouts were subjected to Marsh funnel testing at 30-minute intervals for a period of 3 hours. The tests were all performed in the laboratory with an ambient temperature of 25±1 degrees Celsius. The grouts were agitated periodically during the 3-hour period using a large spoon to simulate the agitation typical of the field handling of the grouts. The test results are shown in Table 6. Each Marsh funnel time reported in the table represents the average of two individual test results. The C75 grout exhibited the least increase in Marsh funnel time over the 3-hour period. After three hours, all of the grouts still satisfied the implementability criterion that the Marsh funnel test result is less than 420 seconds.

Table 6. Influence of grout age on the Marsh funnel time. Marsh funnel times in seconds.

	Grout Product				
	C75	E	S	T	U
Grout Age (hours)	Modified Tank Closure	Enviro-Blend	Salt Stone	Tect HG	US Grout
0.0	67	132	84	113	54
0.5	69	132	104	133	65
1.0	71	139	110	134	69
1.5	69	151	149	139	73
2.0	70	163	153	154	77
2.5	69	166	185	162	77
3.0	71	168	205	193	86

3.2 Interference Tolerance Testing

The materials at the INEEL that are being considered for treatment with these grouts are represented by three materials referred to as interferences. The three interferences are organic sludge, a nitrate salt mixture, and soil from the INEEL. To evaluate any effects that these materials will have on the strength characteristics of the grout, mixtures of grout and interference were prepared using various interference dosages. The compressive strength of three specimens for each grout-interference combination was determined using unconfined compression testing at a specimen age of 14 days. The results of this testing are presented in Table 7. In general, the values presented in Table 6 represent the average of three test results. In four cases, fewer than three test results were available due to testing errors or poor quality samples. These are indicated using the symbols indicated at the bottom of the table. The use of NA in the table indicates that data are not available for these entries. These mix combinations either were too dry and stiff to allow the mixing and fabrication of test specimens, or the strength was so low that the specimens could not be handled without damaging them. The individual compressive strength test results are contained in Appendix D.

Table 7. Average compressive strength (in MPa) for the interference tolerance testing specimen groups.

		Grout Product				
		C75	E	S	T	U
Interference Type	Interference Percentage	Modified Tank Closure	Enviro-Blend	Salt Stone	Tect HG	US Grout
None		52.67	1.03	9.00	43.57	17.80
INEEL Soil	12	40.57	0.43	8.68	28.61	26.86
INEEL Soil	25	41.70	0.18	6.27	25.19	21.36
INEEL Soil	50	17.44	0.30	9.09	13.27	8.81
INEEL Soil	75	NA	NA	2.78	NA	5.55
Nitrate Salts	12	21.86	0.27	4.83	22.33‡	33.10
Nitrate Salts	25	19.89	0.03	2.78	8.23	9.54
Nitrate Salts	50	0.02	NA	0.01	NA	12.50
Nitrate Salts	75	0.72	0.08	0.02	NA	5.99
Organic Sludge	3	50.67	0.92	8.79	29.62	22.59
Organic Sludge	5	42.06	0.91	7.41	25.55	19.84
Organic Sludge	7	42.85	0.70	6.79	19.44	18.23
Organic Sludge	9	41.94	0.72	7.04	18.05‡	21.62
Organic Sludge	12	NA	0.80	6.37	16.18	NA
Organic Sludge	25	NA	NA	3.50	1.41†	NA
Organic Sludge	50	NA	0.36	NA	0.05‡	NA

† indicates that the value represents a single test result.
‡ indicates that the value is the average of two test results.
1 MPa = 145.04 pounds per square inch
NA indicates that data is not available.

3.3 Physical Testing

Several tests were performed to evaluate the physical characteristics of the neat grouts and grouts with interferences. For the neat grouts, the specimens were cured in a special temperature-controlled chamber to simulate the curing temperatures that will likely exist during the first several days after grouting under field conditions. The curing procedure is described in detail in section 2.1.3.

Mixtures of grout and interference were prepared for each interference type. The dosage for each of the interference types was established in consultation with INEEL personnel based on the results of the interference tolerance testing. The interference dosages as percent by weight are 9 percent for the organic sludge, 12 percent for the nitrate salt mixture, and 50 percent for the INEEL soil. The results of the physical testing of the neat grout and the mixtures of grout and interference are presented in the next two sections.

3.3.1 Neat Grout

The results for the physical testing of the neat grout specimens are presented in two tables. Table 8 contains the test results for specific gravity, March funnel time, filtration, maximum set temperature, initial gelation time, final gelation time, and hydraulic conductivity. The test results for specific gravity, Marsh funnel time, pressure filtration, and maximum set temperature are very similar to those reported in the implementability section of the report. This is as expected, since the grout materials and testing procedures are the same in both cases. The minor differences between the two groups of tests are primarily due to normal variations in test results associated with standard laboratory accuracies, slight variations in materials, and typical test operator influences. Within each group of tests there are three individual test results, and the mean and standard deviation values are presented along with the data. The data indicate that these test results are very repeatable when the test specimens come from the same batch of grout as they did for these tests.

Some of the initial gelation and final gelation times reported in Table 8 are noticeably different from those reported in Table 5 for the implementability testing phase of the study. One example is the initial gelation time for the S grout is 1.8 hours as reported in Table 5 and 3.1 hours as reported in Table 8. However, the final gelation times of 8.3 and 8.2 hours for this grout from the two tables are in very close agreement. Based on limited experience with the gelation time tests, it appears that the test results for specimens taken from the same batch of grout are very repeatable. However, the results of tests of the same material prepared and tested on different days are noticeably more variable. This is believed to be due to minor differences in laboratory procedure and minor differences in the proportions of the grout mixtures. For commercially prepared materials, the blending that produces a uniform product on a field scale application may not produce a perfectly uniform product on a laboratory scale sample. For the lab prepared blends, minor errors in measuring and adding the admixtures, which are a small portion of the total mix, can have a significant influence on the rate of stiffening of the grout mixture without having a significant influence on the properties of the final product.

The hydraulic conductivity values for the neat grouts are also reported in Table 8. In general, the two test results for each grout agree fairly well. The C75 grout and the T grout had the lowest hydraulic conductivity values. The hydraulic conductivity values for the S and U grouts were similar and slightly higher than those for the C75 and T grouts. The hydraulic conductivity of the E grout was roughly an order of magnitude higher than any of the other grouts.

The compressive strength and splitting tensile strength data for the neat grout specimens are presented in Table 9. The C75, T and U grouts had the highest compressive strengths, and the E grout had the lowest compressive strength. The strengths of the C75, T, and U grouts are higher than what is generally required for the intended application. This may be beneficial in that it may allow these grouts to tolerate higher interference loading rates while still maintaining the desired compressive strength.

Table 8. Results of all physical testing other than strength testing for the neat grouts.

Test	C75 Modified Tank Closure	E Enviro-Blend	S Salt Stone	T Tect HG	U US Grout
Specific Gravity, Test 1	1.85	1.77	1.60	2.16	1.65
Specific Gravity, Test 2	1.85	1.78	1.60	2.16	1.65
Specific Gravity, Test 3	1.84	1.78	1.60	2.16	1.65
Average Specific Gravity	1.85	1.78	1.60	2.16	1.65
Standard Deviation	0.00	0.00	0.00	0.00	0.00
Marsh Funnel, Test 1 (sec)	62	164	87	129	49
Marsh Funnel, Test 2 (sec)	63	165	97	141	50
Marsh Funnel, Test 3 (sec)	57	166	103	148	53
Average Marsh Funnel (sec)	61	165	96	139	51
Standard Deviation	3	1	7	8	2
Filtration Test, Test 1 ($min^{-0.5}$)	0.087	0.084	0.024	0.008	0.026
Filtration Test, Test 2 ($min^{-0.5}$)	0.080	0.082	0.023	0.008	0.026
Filtration Test, Test 3 ($min^{-0.5}$)	0.084	0.082	0.024	0.008	0.024
Average Filtration Test ($min^{-0.5}$)	0.083	0.083	0.024	0.008	0.025
Standard Deviation	0.003	0.001	0.000	0.000	0.001
Max. Set Temp., Test 1 (ºC)	63	32	29	68	53
Max. Set Temp., Test 2 (ºC)	62	34	28	67	54
Max. Set Temp., Test 3 (ºC)	64	32	28	67	54
Average Max. Set Temp., (ºC)	63	33	28	67	54
Standard Deviation	0.8	0.9	0.5	0.5	0.5
Initial Gelation, Test 1 (hours)	5.5	5.3	3.0	6.2	6.9
Initial Gelation, Test 2 (hours)	5.6	6.0	3.1	6.4	6.9
Initial Gelation, Test 3 (hours)	5.5	4.8	3.2	6.5	6.9
Average Initial Gelation (hours)	5.5	5.4	3.1	6.4	6.9
Standard Deviation	0.1	0.5	0.1	0.1	0.0
Final Gelation, Test 1 (hours)	11.8	22.1	8.4	18.2	9.3
Final Gelation, Test 2 (hours)	11.9	22.2	8.2	18.6	9.5
Final Gelation, Test 3 (hours)	12.0	24.3	8.0	17.9	9.4
Average Final Gelation (hours)	11.9	22.9	8.2	18.2	9.4
Standard Deviation	0.1	1.0	0.2	0.3	0.1
Hydraulic Conductivity, Test 1 (cm/sec)	8.5E-09	1.6E-07	1.2E-08	9.8E-09	1.7E-08
Hydraulic Conductivity, Test 2 (cm/sec)	6.1E-09	1.3E-07	1.6E-08	1.7E-09	1.9E-08
Average Hydraulic Conductivity (cm/sec)	7.3E-09	1.5E-07	1.4E-08	5.8E-09	1.8E-08
Standard Deviation	1.2E-09	1.5E-08	2.0E-09	4.0E-09	1.0E-09

Table 9. Compressive strength and splitting tensile strength values (in MPa) for the neat grouts.

	Grout Product				
	C75	E	S	T	U
Test	Modified Tank Closure	Enviro-Blend	Salt Stone	Tect HG	US Grout
Compressive Strength, Specimen A	51.10	0.71	9.70	51.32	56.74
Compressive Strength, Specimen B	51.30	0.59	10.05	45.27	58.21
Compressive Strength, Specimen C	47.42	0.72	8.48	53.88	65.02
Compressive Strength, Specimen D	44.56	0.69	9.80	54.79	65.03
Compressive Strength, Specimen E	48.99	0.72	9.65	47.73	59.05
Average Compressive Strength	48.68	0.68	9.54	50.60	60.81
Standard Deviation	2.51	0.05	0.55	3.62	3.52
Tensile Strength, Specimen A	4.61	0.09	0.87	5.22	2.29
Tensile Strength, Specimen B	5.76	0.08	1.08	5.23	3.12
Tensile Strength, Specimen C	5.38	0.09	0.59	5.38	4.23
Tensile Strength, Specimen D	4.43	0.10	1.14	4.77	4.56
Tensile Strength, Specimen E	4.17	0.10	0.95		3.32
Average Tensile Strength	4.87	0.09	0.92	5.15	3.50
Standard Deviation	0.60	0.01	0.19	0.23	0.81

1 MPa = 145.04 pounds per square inch

3.3.2 Grout with Interferences

The physical testing of the grouted interference specimens include tests to determine the splitting tensile strength, compressive strength, bulk density and hydraulic conductivity of each grout-interference combination. The interference loading rates were established in consultation with INEEL personnel based on the results of the interference tolerance testing phase of the study. The loading rates are 12 percent for the nitrate salt mixture, 9 percent for the organic sludge, and 50 percent for the INEEL soil. The loading rates are weight percentages based on the total weight of the grout plus the interference.

The tensile strength data and the compressive strength data are presented in Tables 10 and 11, respectively. In all cases, the grout-interference combinations involving the C75 grout had the highest compressive strength and the highest splitting tensile strength. For the compressive strength values, the specimens containing the U grout was the next strongest followed by those containing the T grout. The compressive strengths of the specimens containing the S and E grouts were significantly less than those for the specimens containing the other three grouts. The compressive strengths of the specimens containing the E grout were particularly low relative to the specimens containing the other grouts. The relative strengths for the splitting tensile strength testing were very similar to those for the compressive strength testing except that for the splitting tensile strength testing, the specimens containing the T grout were slightly stronger than those containing the U grout. This is the opposite of what was observed for the compressive strength testing.

Table 10. Splitting tensile strength values (in MPa) for mixtures of each grout with the various interferences.

		Grout Product				
		C75	E	S	T	U
Test	Interference Amount and Type	Modified Tank Closure	Enviro-Blend	Salt Stone	Tect HG	US Grout
Specimen A	12% Nitrate Salts	2.57	0.03	0.58	2.16	1.77
Specimen B	12% Nitrate Salts	1.78	0.03	0.48	2.04	1.75
Specimen C	12% Nitrate Salts	1.70	0.03	0.66	1.21	1.26
Specimen D	12% Nitrate Salts	2.87	0.03	0.72	2.17	1.31
Specimen E	12% Nitrate Salts	2.85	0.03	0.55	2.59	1.81
Average	12% Nitrate Salts	2.35	0.03	0.60	2.03	1.58
Std. Deviation	12% Nitrate Salts	0.51	0.00	0.08	0.45	0.24
Specimen A	9% Organic Sludge	3.55	0.13	0.67	2.39	1.29
Specimen B	9% Organic Sludge	3.36	0.12	0.64	2.28	1.36
Specimen C	9% Organic Sludge	3.54	0.12	0.73	1.66	1.19
Specimen D	9% Organic Sludge	3.56	0.13	0.69	2.15	1.05
Specimen E	9% Organic Sludge	3.28	0.12	0.68	2.21	1.14
Average	9% Organic Sludge	3.46	0.12	0.68	2.14	1.21
Std. Deviation	9% Organic Sludge	0.12	0.00	0.03	0.25	0.11
Specimen A	50% INEEL Soil	2.12	0.03	0.92	2.16	1.59
Specimen B	50% INEEL Soil	2.88	0.02	0.63	2.20	1.77
Specimen C	50% INEEL Soil	2.43	0.02	1.11	1.95	1.33
Specimen D	50% INEEL Soil	2.48	0.01	0.99	2.26	1.55
Specimen E	50% INEEL Soil	2.30	0.02	0.93	2.09	1.39
Average	50% INEEL Soil	2.44	0.02	0.92	2.13	1.53
Std. Deviation	50% INEEL Soil	0.25	0.01	0.16	0.11	0.16

1 MPa = 145.04 pounds per square inch

Table 11. Compressive strength values (in MPa) for mixtures of each grout with the various interferences.

		Grout Product				
		C75	E	S	T	U
Test	Interference Amount and Type	Modified Tank Closure	Enviro-Blend	Salt Stone	Tect HG	US Grout
Specimen A	12% Nitrate Salts	34.87	0.19	4.56	20.92	30.09
Specimen B	12% Nitrate Salts	29.21	0.19	4.28	15.55	30.19
Specimen C	12% Nitrate Salts	28.96	0.17	4.21	17.36	32.96
Specimen D	12% Nitrate Salts	43.25	0.19	4.45	10.73	24.28
Specimen E	12% Nitrate Salts	35.50	0.19	4.50	17.60	20.09
Average	12% Nitrate Salts	34.36	0.19	4.40	16.43	27.52
Std. Deviation	12% Nitrate Salts	5.22	0.01	0.13	3.34	4.67
Specimen A	9% Organic Sludge	37.93	0.79	6.71	13.70	21.70
Specimen B	9% Organic Sludge	37.06	0.79	6.99	14.00	23.36
Specimen C	9% Organic Sludge	34.18	0.71	7.03	13.41	22.09
Specimen D	9% Organic Sludge	36.76	0.85	7.17	13.75	19.60
Specimen E	9% Organic Sludge	40.28	0.88	7.18	13.46	17.51
Average	9% Organic Sludge	37.24	0.80	7.02	13.66	20.85
Std. Deviation	9% Organic Sludge	1.97	0.06	0.17	0.21	2.06
Specimen A	50% INEEL Soil	16.19	0.28	7.70	12.63	17.60
Specimen B	50% INEEL Soil	22.77	0.31	7.10	13.07	16.58
Specimen C	50% INEEL Soil	16.38	0.23	7.53	14.53	16.53
Specimen D	50% INEEL Soil	16.82	0.21	7.32	12.92	18.63
Specimen E	50% INEEL Soil	18.73	0.19	7.24	15.02	18.04
Average	50% INEEL Soil	18.18	0.24	7.38	13.63	17.48
Std. Deviation	50% INEEL Soil	2.47	0.04	0.21	0.95	0.82

1 MPa = 145.04 pounds per square inch

The density values for the grout-interference combinations are presented in Table 12. The three values for each combination are in very close agreement with each other. For each particular grout, the densities tend to be the lowest for the organic sludge mixture, and highest for the INEEL soil mixture. This is as expected due to the differences in the densities of the various interferences. For a particular interference, the density of the grout-interference mixture is correlated to the density of the grout involved in the mixture, as is expected.

Table 12. Bulk density of the grout-interference mixtures presented in grams per cubic centimeter.

		Grout Product				
		C75	E	S	T	U
Test	Interference Amount and Type	Modified Tank Closure	Enviro-Blend	Salt Stone	Tect HG	US Grout
Specimen A	12% Nitrate Salts	1.89	1.86	1.65	2.17	1.72
Specimen B	12% Nitrate Salts	1.91	1.89	1.64	2.17	1.71
Specimen C	12% Nitrate Salts	1.91	1.88	1.66	2.16	1.69
Average	12% Nitrate Salts	1.90	1.88	1.65	2.17	1.71
Std. Deviation	12% Nitrate Salts	0.01	0.01	0.01	0.00	0.01
Specimen A	9% Organic Sludge	1.81	1.73	1.55	2.02	1.65
Specimen B	9% Organic Sludge	1.79	1.75	1.55	2.02	1.65
Specimen C	9% Organic Sludge	1.80	1.73	1.56	2.01	1.65
Average	9% Organic Sludge	1.80	1.74	1.55	2.02	1.65
Std. Deviation	9% Organic Sludge	0.01	0.01	0.00	0.00	0.00
Specimen A	50% INEEL Soil	1.97	1.91	1.82	2.14	1.84
Specimen B	50% INEEL Soil	1.96	1.92	1.81	2.14	1.84
Specimen C	50% INEEL Soil	1.97	1.92	1.82	2.15	1.83
Average	50% INEEL Soil	1.97	1.92	1.82	2.14	1.84
Std. Deviation	50% INEEL Soil	0.00	0.00	0.00	0.00	0.00

The hydraulic conductivity values for the grout-interference combinations are presented in Table 13. The most obvious conclusions from the hydraulic conductivity data are that the hydraulic conductivity values for the C75, S, T, and U grouts mixed with the various interferences are fairly similar, and that the hydraulic conductivity values for the E grout tend to be higher (more permeable) than those for the other four grouts. For the mixtures involving the nitrate salt interference, the lowest hydraulic conductivity values were reported for the T and U grouts. For the specimens containing the organic sludge the lowest hydraulic conductivity value was recorded for the C75 and T grouts. For the specimens containing the INEEL soil, the lowest hydraulic conductivity value was recorded for the C75 grout. The E grout performed noticeably better with the organic sludge than it did with either the nitrate salt or the INEEL soil.

Table 13. Hydraulic conductivity values (cm/sec) for mixtures of each grout with the various interferences.

		Grout Product				
		C75	E	S	T	U
Test Specimen	Interference Amount and Type	Modified Tank Closure	Enviro-Blend	Salt Stone	Tect HG	US Grout
Specimen A	12% Nitrate Salts	5E-07	9E-06	2E-08	6E-09	7E-09
Specimen B	12% Nitrate Salts	7E-08	6E-06	2E-08	2E-08	2E-08
Average	12% Nitrate Salts	3E-07	8E-06	2E-08	1E-08	1E-08
Std. Deviation	12% Nitrate Salts	2E-07	2E-06	0E+00	7E-09	7E-09
Specimen A	9% Organic Sludge	2E-09	7E-08	4E-08	5E-09	1E-08
Specimen B	9% Organic Sludge	4E-09	5E-08	2E-08	1E-09	2E-08
Average	9% Organic Sludge	3E-09	6E-08	3E-08	3E-09	2E-08
Std. Deviation	9% Organic Sludge	1E-09	1E-08	1E-08	2E-09	5E-09
Specimen A	50% INEEL Soil	6E-09	7E-07	8E-08	2E-08	3E-09
Specimen B	50% INEEL Soil	1E-08	1E-06	8E-08	8E-09	2E-08
Average	50% INEEL Soil	8E-09	9E-07	8E-08	1E-08	1E-08
Std. Deviation	50% INEEL Soil	2E-09	2E-07	0E+00	6E-09	9E-09

3.4 Chemical Testing

ANS-16.1 is a semi-dynamic test; that is, the leachant is sampled and replaced periodically for a total of 90 days. The test method is applicable to any material that does not degrade, deform, or change its leaching mechanism at the temperatures used in the test. In this report, the results of the ANS 16.1 leaching test were fitted to a semi-empirical mathematical model based on simple leaching rate mechanisms, which permitted the evaluation of an apparent diffusion coefficient and leachability index, thus providing a measure of contaminant mobility in the solidified waste. If less than 20% of a species is leached from a uniform, regularly shaped solid, then its leaching behavior (if diffusion controlled) approximates that of a semi-infinite medium. The mass-transport equation permits the calculation of an "effective coefficient" by the expression (1).

$$De = \pi\left[\frac{an/A_0}{(\Delta t)_n}\right]^2\left[\frac{V}{S}\right]^2 T$$

where

De	=	Effective Diffusivity, cm^2/s,
V	=	Volume of the Specimen, cm^3,
S	=	Geometric surface area of the specimen as calculated from measured dimensions (cm^2), and (2)

$$T = \left[\frac{1}{2}(t_n^{1/2} + t_{n-1}^{1/2}\right]^2$$

Leaching time (T) represents the "mean time" of the leaching interval. Test were conducted on both neat grout and grout with interferences. The leach index (LI) is equal to – log (D_e). This information will provide an estimate of the long-term physical and chemical durability of the grout material and an estimate of the rate of diffusion of contaminant species from the grout matrix. The release rate of calcium, aluminum, and silicon provide a measure of the dissolution rate of the grout matrix and may be used to estimate the time the grout will provide physical stability to the waste-site, whereas strontium and nitrate release rates provide an estimate for contaminant species.

The solubility of hazardous waste constituents is primarily a function their chemical environment, which can be characterized by the pH (acid-base property) and oxidation-reduction potential (Eh) of the surrounding medium. The pH and Eh of the grout formulations was measured in the leachate for the ANS 16.1 leaching testing for both neat grouts and grouts with interferences. The Eh and pH are indicative of the buffered chemical environment produced by the grout monolith and solubility of the encapsulated waste constituents.

3.4.1 Neat Grout

In order to determine the diffusion of elements present in the grout, a digestion was performed and the results are shown in Table 14.

The average leach index (n=3) for each grout is shown in Table 15(a) and Table 15(b) shows pH and Eh ranges for each grout during testing (Appendix A has individual sample test results). All grouts exhibited relatively high leach indexes (i.e. low leaching potential) for constituents in the grout (aluminum, calcium, and silicon), ranging from 9.8 to 14.5. It is difficult to make direct comparisons between the grouts without knowledge of specific chemical compositions; however, the values of Table 14 and Table 15(a) could be used to estimate the time required for individual elements to completely leach. The pH range difference for each grout was relatively consistent, varying by 1.3 pH units for the U and C75 grouts (smallest range) and 1.9 pH units for the E grout (largest range). Examination of absolute values for pH also showed that all grouts operated at approximately similar pH values. C75 and S grout exhibited the narrowest Eh range, with T grout showing the widest range. These values do give some indication over a 90-day period of the chemical equilibrium of the neat grout.

Table 14. Element Concentration Digestion Determination for Each Grout

		Element (mg/g)	
Grout	Al	Si	Ca
U	7.79	10.69	37.01
T	7.28	14.87	107.56
E	4.88	19.08	4.31
C75	6.91	8.04	91.64
S	16.48	5.25	46.48

Note: Neat grouts spiked with strontium (Sr = 0.593 mg/g) and nitrate (NO_3^{-2} = 0.614 mg/g).

Assessment of strontium leach index values again shows low leaching potential with the E grout exhibiting the lowest potential and the other four grouts behaving quite similar. This was not unexpected, as the E grout is believed to contain significant phosphate that could complex with strontium. Nitrate is likely a better estimate of a conservative tracer and shows T, C75, and S grouts (statistically these three overlap) to have lower leaching potential than the U and E grouts.

Table 15(a). Neat grout average leach index (n=3) results for Sr, Al, Ca, Si, and NO_3^-.

Grout	Sr	Al	Ca	Si	NO_3^-
U	10.6 ± 0.9	11.1 ± 0.4	9.8 ± 0.9	10.2 ± 0.7	9.2 ± 0.3
T	10.1 ± 0.3	12.3 ± 0.6	10.1 ± 0.5	11.1 ± 0.5	11.0 ± 0.7
E	12.8 ± 1.2	14.5 ± 1.6	9.8 ± 0.3	14.2 ± 1.5	8.8 ± 0.2
C75	10.0 ± 0.5	12.2 ± 0.8	10.5 ± 0.5	10.7 ± 1.1	10.4 ± 0.6
S	10.2 ± 0.6	12.6 ± 0.9	10.5 ± 1.0	10.2 ± 0.9	10.8 ± 0.8

Results reported ± one standard deviation.

Table 15(b). pH and Eh range during neat grout leach tests (n=3).

Grout	pH Range	Eh Range (mV)
U	9.9 to 11.2	130 to 391
T	9.6 to 11.4	66 to 380
E	8.7 to 10.8	122 to 378
C75	10.3 to 11.6	172 to 313
S	9.5 to 11.0	192 to 301

3.4.2 Grout with Interferences

The waste interference composition for the nitrate salt waste is shown in Table 16.

Table 16. Material proportions for the nitrate salt interference mixture.

Ingredient	Percent by Weight
Sodium Nitrate	60.0
Potassium Nitrate	30.0
Sodium Sulfate	5.0
Sodium Chloride	5.0

The average leach index (n=3) for strontium for each grout as a function of interference is shown in Table 17 (Appendix B has individual sample test results). The average leach index for all grouts ranged from 10.3 to 12.6, indicating low leaching potential for strontium in the presence of organic sludge, nitrate salt, and INEEL soil at the designated loadings. Interestingly, the measured leach indices are comparable for each neat grout, indicating little effect of the interference on strontium release and perhaps that nitrate release may be a better measure to distinguish grout performance.

Table 17. Average leach index (n=3) results for strontium as a function of interference.

Grout	9 wt% Organic Sludge	12 wt% Nitrate Salt	50 wt% INEEL Soil
U	10.8 ± 0.7	11.6 ± 0.5	11.4 ± 0.8
T	10.4 ± 0.6	10.6 ± 0.7	10.5 ± 0.9
E	12.1 ± 0.7	12.2 ± 0.9	12.6 ± 0.9
C75	10.3 ± 0.6	10.9 ± 0.6	10.6 ± 0.5
S	10.4 ± 0.4	10.4 ± 0.4	10.5 ± 0.5

Results reported ± one standard deviation.

Figure 6 shows an example of S grout after 90 days. Clearly, the sample does not have any visible deterioration. Durability testing should be conducted, however, to fully evaluate the overall performance of the interference samples exposed to water and other environmental conditions.

3.5 WAXFIX Boron Distribution

The purpose of this evaluation was to measure boron distribution in the WAXFIX grout. Multiple formulations were provided by the manufacturer over the duration of the project and prepared according to their specifications. Table 18 shows measured boron concentrations as a function of relative depth in a sample prepared using the initial WAXFIX preparation steps and chemicals. Based on these results the boron clearly did not distribute well in the sample. Boron settling in the WAXFIX is an undesirable property in that introduction of the WAXFIX in a pit containing Pu-239 and U-233 and U-235 raises the possibility of an uncontrolled criticality. The test plan called for 1 g/l of B-10, the effective boron concentration that has excellent neutron absorption properties and a large safety factor in criticality calculations.

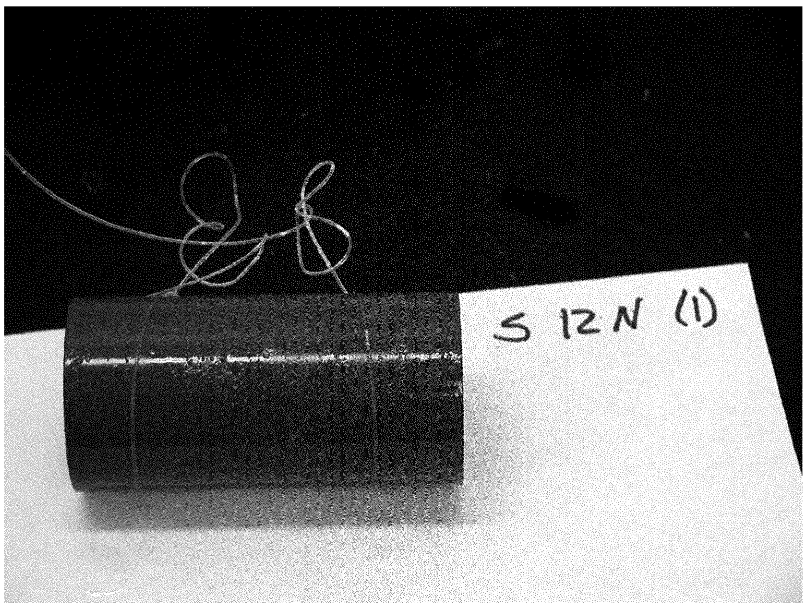

Figure 6. S grout sample (containing 12% nitrate salt waste) after leach testing (90 days).

Additional surfactants and process modifications failed to remedy the settling problem. Figure 7 displays the poor settling that occurred with the final formulation provided by the manufacturer, confirming actual TCLP measurements (e.g. Table 18). These results suggest that a completely different boron introduction scheme be devised to allow proper distribution in the sample.

Table 18. Boron distribution and pH measurement for WAXFIX grout after TCLP.

Sample Location	Boron (mg/L)	pH
Top	18.5	2.9
Middle	43.5	3.0
Bottom	316.0	5.1

Note: Top refers to the top one-third depth and Bottom refers to the lower one-third depth.

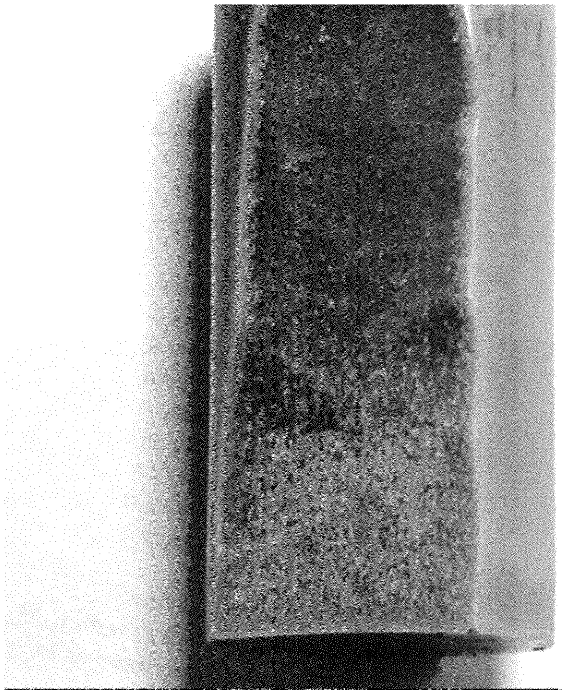

Figure 7. WAXFIX sample (based on final modified WAFIX formulation) showing poor boron distribution.

3.6 Grout-Organic Sludge Encapsulation

3.6.1 Microencapsulation

This test involved evaluation of organic diffusion (TCE, TCA, PCE, and CTET) from the grout after mixing with organic sludge. Based on earlier physical and chemical testing results, only the C75, T, and U grouts were selected for this evaluation. Figure 8 shows a C75 grout sample after mixing and curing. Table 19 is a summary of the gas phase concentrations and mass percentage for each compound over the course of 90 days. Recall that different masses of organics were in the mixtures (Table 4), therefore, the mass percentage data is better for comparison purposes.

There are several basic trends observed for all three grouts during the course of the experiment. First, the mass percentage typically stabilized over the course of time. This would be expected as there is likely a rapid near-surface release of organic followed by both equilibration and diffusion transfer from the solid. Second, there was not much difference between grouts in the final measured gas phase values and the values were all approximately less than 0.06%, indicating the ability of all three to capture these highly volatile organic compounds. Sample dimensions are provided to enable specific release rate calculations, however, this test procedure represents a dynamic situation and may require longer than 90 days to reach equilibrium.

3.6.2 Macroencapsulation

This test involved evaluation of organic diffusion (TCE, TCA, PCE, and CTET) from a plug of organic sludge placed inside of the grout and capped on the end. Similar to the microencapsulation test, only the C75, T, and U grouts were selected for this evaluation. Table 20 is a summary of the gas phase concentrations and mass percentage for each compound over the course of 90 days. Given that the volume of the plug was the same for all three grouts, they contained equal amounts of organic sludge. Therefore, comparison of mass percentage data for this test should be a fair comparison. Figure 9 shows a C75 grout sample, with some cracking evident in the plug area and similar cracks were observed on the U and T grouts although not as severe. This cracking was not evident until well after the testing was initiated (i.e. greater than 30 days) and was most likely caused by differential curing between the seal material and the core cylinder. Given the observed cracks in the sample that appeared over time, strict data interpretation and release calculations should be made with caution. Based simply on the measured data, however, the grouts again show low release of the organic compounds (all less than 0.16% after 90 days). Plug dimensions are available to enable specific release rate calculations through the grout matrix, however, the test procedure should be modified to continuously measure diffusion across a fixed length and overcome the inherent problem of cracking that was observed in this study.

Figure 8. C75 grout microencapsulation sample.

Table 19. Gas phase concentration and mass percentage data for microencapsulation test.

(a) C75 Grout

Day	CTET (mg/L)	PCE (mg/L)	TCE (mg/L)	TCA (mg/L)	CTET (%)	PCE (%)	TCE (%)	TCA (%)
0	9.55	2.97	7.39	0.04	0.021	0.023	0.064	BDL
10	135.97	26.36	49.78	0.68	0.299	0.205	0.431	0.005
20	9.16	5.30	7.15	BDL	0.020	0.041	0.062	BDL
30	11.32	10.86	9.13	BDL	0.025	0.084	0.079	BDL
40	10.37	5.10	7.43	BDL	0.023	0.040	0.064	BDL
50	9.10	7.82	7.94	BDL	0.020	0.061	0.069	BDL
60	7.63	3.95	5.83	BDL	0.017	0.031	0.050	BDL
70	6.34	4.92	6.70	BDL	0.014	0.038	0.058	BDL
80	8.08	5.29	6.44	BDL	0.018	0.041	0.056	BDL
90	7.82	5.30	6.72	BDL	0.017	0.041	0.058	BDL

Table 19. (continued)

(b) T Grout

Day	CTET (mg/L)	PCE (mg/L)	TCE (mg/L)	TCA (mg/L)	CTET (%)	PCE (%)	TCE (%)	TCA (%)
0	6.01	2.01	6.38	0.23	0.012	0.014	0.049	BDL
10	25.01	7.67	22.24	0.30	0.048	0.053	0.170	BDL
20	14.17	6.65	11.97	0.13	0.027	0.046	0.091	0.001
30	10.20	6.21	10.39	BDL	0.020	0.043	0.079	BDL
40	12.95	5.40	10.87	BDL	0.025	0.037	0.083	BDL
50	11.14	7.90	11.36	BDL	0.022	0.054	0.087	BDL
60	9.91	4.55	9.10	BDL	0.019	0.031	0.070	BDL
70	6.72	4.85	9.12	BDL	0.013	0.033	0.070	BDL
80	6.25	4.28	7.56	BDL	0.012	0.029	0.058	BDL
90	6.58	4.57	7.80	BDL	0.013	0.031	0.060	BDL

(c) U Grout

Day	CTET (mg/L)	PCE (mg/L)	TCE (mg/L)	TCA (mg/L)	CTET (%)	PCE (%)	TCE (%)	TCA (%)
0	5.90	6.33	9.59	BDL	0.014	0.054	0.092	BDL
10	9.21	6.07	6.67	0.19	0.022	0.052	0.063	0.001
20	4.68	5.09	4.34	BDL	0.011	0.044	0.041	BDL
30	6.30	13.14	6.37	BDL	0.015	0.113	0.061	BDL
40	1.94	2.28	3.98	BDL	0.005	0.020	0.038	BDL
50	2.24	4.18	2.98	BDL	0.005	0.036	0.028	BDL
60	1.92	2.26	2.05	BDL	0.005	0.019	0.020	BDL
70	1.45	2.55	2.43	BDL	0.004	0.022	0.023	BDL
80	1.66	2.54	2.25	BDL	0.004	0.022	0.021	BDL
90	1.52	2.53	2.27	BDL	0.004	0.022	0.022	BDL

Notes:

1. Day 10 data, particularly for C75 grout, appear unusually high (i.e. potential analytical or sampling error).
2. All values reported are average of three (3) separate samples/bottles.
3. BDL = Below Detection Limit
4. Sample size of 7.62 cm diameter by 6.35 cm height and air volume of 15.42 mL.

Table 20. Gas phase concentration and mass percentage data for macroencapsulation test.

(a) C75 Grout

Day	CTET (mg/L)	PCE (mg/L)	TCE (mg/L)	TCA (mg/L)	CTET (%)	PCE (%)	TCE (%)	TCA (%)
10	61.88	13.12	26.95	BDL	0.151	0.111	0.281	BDL
20	53.38	21.42	25.63	0.05	0.130	0.181	0.268	0.001
30	33.08	10.73	16.54	6.45	0.081	0.091	0.173	0.058
40	23.28	13.40	13.52	8.31	0.057	0.113	0.141	0.074
50	14.52	19.33	11.38	8.14	0.035	0.163	0.119	0.073

Table 20. (continued)

(a) C75 Grout

Day	CTET (mg/L)	PCE (mg/L)	TCE (mg/L)	TCA (mg/L)	CTET (%)	PCE (%)	TCE (%)	TCA (%)
60	5.76	14.02	7.98	6.80	0.014	0.118	0.083	0.061
70	3.33	9.67	4.64	4.85	0.008	0.082	0.048	0.043
80	2.43	16.74	4.16	5.15	0.006	0.141	0.043	0.046
90	0.83	18.74	3.60	4.45	0.002	0.158	0.038	0.040

(b) T Grout

Day	CTET (mg/L)	PCE (mg/L)	TCE (mg/L)	TCA (mg/L)	CTET (%)	PCE (%)	TCE (%)	TCA (%)
10	2.06	2.71	22.24	BDL	0.005	0.023	0.232	BDL
20	1.24	0.94	2.17	BDL	0.003	0.008	0.023	BDL
30	7.97	2.69	5.62	1.00	0.019	0.023	0.059	0.009
40	1.19	0.75	1.44	0.33	0.003	0.006	0.015	0.003
50	0.93	0.92	1.38	0.39	0.002	0.008	0.014	0.003
60	0.76	0.62	1.29	0.28	0.002	0.005	0.013	0.002
70	0.19	0.37	1.54	BDL	0.001	0.003	0.016	BDL
80	1.03	0.85	1.65	0.40	0.003	0.007	0.017	0.004
90	0.94	0.91	1.98	0.44	0.002	0.008	0.021	0.004

(c) U Grout

Day	CTET (mg/L)	PCE (mg/L)	TCE (mg/L)	TCA (mg/L)	CTET (%)	PCE (%)	TCE (%)	TCA (%)
10	15.48	4.57	8.62	BDL	0.038	0.039	0.090	BDL
20	11.06	2.31	5.66	0.22	0.027	0.020	0.059	0.002
30	13.14	2.94	6.35	0.11	0.032	0.025	0.066	0.001
40	11.04	2.43	6.01	1.11	0.027	0.021	0.063	0.010
50	13.52	4.45	7.38	1.51	0.033	0.038	0.077	0.014
60	9.37	5.26	7.08	7.32	0.023	0.044	0.074	0.065
70	10.12	2.31	6.29	1.28	0.025	0.020	0.066	0.011
80	20.59	5.99	11.53	2.96	0.050	0.051	0.120	0.027
90	15.67	5.63	11.90	2.56	0.038	0.048	0.124	0.023

Notes:
1. All values reported are average of three (3) separate samples/bottles.
2. BDL = Below Detection Limit
3. Sample size of 7.62 cm diameter by 6.35 cm height and air volume of 15.42 mL.

Figure 9. C75 grout macroencapsulation sample showing some small cracks where plug was sealed.

4. CONCLUSIONS AND RECOMMENDATIONS

Based on the results of this study, the following conclusions are made:

1. Neat grout samples show low leaching potential of grout constituents (aluminum, calcium, and silicon), with average leach indexes all greater than 9.8 and as high as 14.5. The pH of these samples was also relatively stable (range of 1-2 pH units) over the course of 90 days.

2. Strontium release from neat grout samples was low (leach indexes all greater than 10.0) and nitrate exhibited low leaching potential (leach indexes all greater than 8.8). T, C75, and S grouts (statistically these three overlap) have lower leaching potential than the U and E grouts.

3. Strontium release from grout samples containing waste interferences (organic sludge, nitrate salt, and INEEL soil) was low (leach indexes all greater than 10.3) and samples did not noticeably deteriorate over the course of 90 days in water.

4. Under multiple conditions specified by the manufacturer, boron settling in the WAXFIX grout is significant and problematic.

5. Gas phase release of volatile organic compounds from U, T, and C75 grouts under a microencapsulation test procedure showed less than 0.06% release after 90 days, indicating the ability of all three grouts to potentially encapsulate volatile organic compounds. There was also minimal difference in measurements of the three grouts.

6. Gas phase release of volatile organic compounds from U, T, and C75 grouts under a macroencapsulation test procedure showed less than 0.16% release after 90 days, indicating the ability of all three grouts to potentially encapsulate volatile organic compounds. There was,

however, cracking evident in the sample plug area after approximately 30 days that could have impacted measured gas-phase release concentrations.

7. All of the grouts satisfied each of the implementability criteria with the exception of the S grout having an initial gelation time that was slightly less than the 2-hour criterion. However, later testing of the neat grouts indicated that the S grout did satisfy the initial gelation criterion.

8. Interference tolerance test results indicate that the organic sludge and the nitrate salt mixture have a significant effect on the strength of the grout-interference mixtures. The higher dosages evaluated in the study proved to be impractical for field implementation. This was either due to the extreme impact on the strength of the resulting specimen or due to difficulty in mixing the specimens at the higher interference loading rates.

9. For the neat grouts, the C75 grout and the T grout had the lowest hydraulic conductivity values.

10. For the neat grouts the C75, T and U grouts had the highest compressive strengths, and the E grout had the lowest compressive strength.

11. For the interference loadings selected for detailed evaluation, the grout-interference combinations involving the C75 grout had the highest compressive strength and the highest splitting tensile strength in all cases.

12. For the interference loadings selected for detailed evaluation, the hydraulic conductivity values for the C75, S, T, and U grouts mixed with the various interferences were fairly similar, and that the hydraulic conductivity values for the E grout were higher (more permeable) than those for the other four grouts.

Based on this study results and conclusions, the following recommendations are made:

1. Nitrate release may be a better measure to distinguish grout-leaching performance and should be utilized in future leaching tests.

2. A new boron distribution process is needed if WAXFIX is to be a candidate grout for in-situ mixed waste applications.

3. The freeze-thaw performance and durability of these grouts in the presence of waste interferences should be evaluated.

4. Modifications and/or other test procedures should be implemented to fully measure the encapsulation and release of organic compounds from grout samples.

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